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## TRANSLATION FROM JAPANESE

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[ Title of the Invention ]            Annular Seal and Method for Manufacturing Same

[ Claims ]

[ Claim 1 ]    An annular seal, characterized in that a sheet or film made of extended porous polytetrafluoroethylene is structured in multiple layers in the radial direction.

[ Claim 2 ]    An annular seal, characterized in that a strip or film made of extended porous polytetrafluoroethylene is wound and laminated spirally or concentrically with the retention of an air core.

[ Claim 3 ]    An annular seal as defined in Claim 1 or 2, wherein at least one fluid permeation inhibitor layer is formed.

[ Claim 4 ]    An annular seal as defined in Claim 3, wherein said fluid permeation inhibitor layer is composed of compact polytetrafluoroethylene.

[ Claim 5 ]    An annular seal as defined in Claim 4, wherein said compact polytetrafluoroethylene is obtained by squeezing the pores of extended porous polytetrafluoroethylene.

[ Claim 6 ]    An annular seal as defined in Claims 1 through 5, wherein said sheet or film made of extended porous polytetrafluoroethylene is bonded and laminated by calcining.

[ Claim 7 ]    An annular seal as defined in Claims 1 through 5, wherein said sheet or film made of extended porous polytetrafluoroethylene is bonded and laminated by means of an adhesive.

[ Claim 8 ]    A method for manufacturing an annular seal as defined in Claim 3, wherein said manufacturing method is characterized by comprising a step in which a strip or film made of extended porous polytetrafluoroethylene is wound and laminated a prescribed number of times, and a step in which a strip or film constituting a fluid permeation inhibitor layer is wound and laminated.

[ Claim 9 ] A method for manufacturing an annular seal as defined in Claim 3, wherein said manufacturing method is characterized by comprising a step in which a strip or film made of extended porous polytetrafluoroethylene is wound and laminated a prescribed number of times, and a step in which the strip or film made of extended porous polytetrafluoroethylene and obtained by the coating or lamination of a material constituting a fluid permeation inhibitor layer is wound and laminated a prescribed number of time.

[ Claim 10 ] A method for manufacturing an annular seal as defined in Claim 5, wherein said manufacturing method is characterized in that said fluid permeation inhibitor layer composed of compact polytetrafluoroethylene is obtained by squeezing the pores of said strip or film made of extended porous polytetrafluoroethylene at least over a distance corresponding to a single turn.

[ Claim 11 ] A method for manufacturing an annular seal, wherein calcining is performed following the completion of the step defined in any of Claims 8 through 10.

[ Claim 12 ] A method for manufacturing an annular seal as defined in Claim 1 or 2, wherein said manufacturing method is characterized in that

a cylinder made of extended porous polytetrafluoroethylene is formed by a process in which a strip or film made of extended porous polytetrafluoroethylene is wound and laminated helically with the retention of an air core, or a wide strip or film made of extended porous polytetrafluoroethylene is wound and laminated spirally or concentrically with the retention of an air core; and

said cylinder is cut into prescribed lengths in the direction intersecting the longitudinal direction of said cylinder.

[ Claim 13 ] A method for manufacturing an annular seal as defined in any of Claims 8 through 10, wherein said manufacturing method is characterized in that

a cylinder having a fluid permeation inhibitor layer is formed by a process in which said strip or film is wound and laminated helically with the retention of an air core, or said wide strip or film is used to perform winding and lamination spirally or concentrically with the retention of an air core; and

said cylinder is cut into prescribed lengths in the direction intersecting the longitudinal direction of said cylinder.

[ Claim 14 ] A method for manufacturing an annular seal as defined in Claim 12 or 13, wherein said manufacturing method involves cutting said cylinder after it has been calcined.

[ Detailed Description of the Invention ]

[ 0001 ]

[ Field of Industrial Utilization ]

The present invention relates to an annular seal for sealing piping flanges, shaft or pin lubrication points, or the like, and to a method for manufacturing such a seal. For example, [the present invention] relates to an annular seal for sealing hard disk drives and other types of precision electronic equipment requiring dustproofness, waterproofness, and drip-proofness, or for sealing the joints of piping that transports corrosive fluids, such as the piping used in equipment for producing pharmaceuticals, foodstuffs, and the like; and to a method for manufacturing such a seal.

[ 0002 ]

[ Prior Art ]

Seals made of materials that generate only small amounts of dust are used in hard disk drives and other types of precision electronic equipment because superclean spaces must be maintained inside such equipment. In addition, seals highly resistant to corrosion are used for the joints of piping systems carrying pharmaceuticals, foodstuffs, and other corrosive fluids. Polytetrafluoroethylene seals are examples of seals composed of materials that generate only small amounts of dust and are highly resistant to corrosion.

[ 0003 ]

A seal for a fastening member must adhere securely to the irregularities of the surface (hereinafter referred to as "fastening surface") in contact with this seal in order to satisfy stringent sealing requirements. However, unextended polytetrafluoroethylene manufactured by sintering (hereinafter referred to as "sintered PTFE") is hard and thus does not conform readily to the rough fastening surface, making it impossible to obtain adequate sealing characteristics unless the fastening torque is increased substantially.

Another drawback is that when adequate fastening pressure cannot be applied to the fastening member, as is the case with a ceramic component, glass substrate, or the like, a PTFE seal fails to adhere sufficiently well to the fastening surface, and fluid leaks through the interface between the seal and the fastening surface (hereinafter this phenomenon will be referred to as "interface leakage").

[ 0004 ]

Attention has been attracted in recent years to seals made of extended porous polytetrafluoroethylene (hereinafter abbreviated as "ePTFE") that adhere better to fastening surfaces and can prevent interface leakage with a comparatively weak fastening force. For example, Japanese Laid-Open Utility Model Application 3-89133 describes annular seals manufactured from ePTFE by a process in which a sheet obtained by laminating and integrating ePTFE films in a prescribed thickness is punched out to produce annular shapes. The term "ePTFE film" refers to a porous PTFE film fashioned into a fibrous structure by stretching. The fibrous structure of ePTFE allows an annular seal made of ePTFE to better conform to the microscopic irregularities of the fastening surface. In addition, [ePTFE] is softer than sintered PTFE, and can therefore be deformed in the thickness direction of the seal, making it possible to reduce interface leakage and to improve the sealing properties.

[ 0005 ]

[ Problems Which the Invention Is Intended to Solve ]

As shown in Figure 13, a manufacturing method in which sheets are punched out is uneconomical because the portions that remain after the annular components 31 have been punched out from an ePTFE sheet 30 cannot be used and are discarded. Another drawback is that the inherent strength and elasticity of ePTFE cannot be achieved because the polymer chains of ePTFE are broken during punching when fabrication involves punching out ePTFE sheets.

[ 0006 ]

Methods in which ePTFE tubes are fabricated by extrusion molding and these tubes are cut in the direction intersecting the longitudinal direction to form annular seals have been proposed as methods that allow annular seals to be fabricated with maximum use of materials. However, limitations are imposed on the drawdown ratio of extrusion molding because of the need to secure adequate particle orientation. For

this reason, the dimensions of the annular seals that can be manufactured by extrusion molding are limited to an inside diameter (d) of 0.5–50 mm, an outside diameter (D) of 0.7–90 mm, and a width  $((D - d) \div 2)$  of about 0.1–20 mm. In other words, it is fairly difficult to manufacture annular seals for use in piping joints whose outside diameter exceeds 90 mm by a manufacturing method involving extrusion molding. On the other hand, a need exists for manufacturing annular seals by reduced-waste methods because the amount of waste generated by the manufacturing methods involving seal punching increases with an increase in diameter. In addition, extrusion molding produces annular seals made of uniaxial ePTFE stretched in the longitudinal direction of the cylinder (in the thickness direction of the seal) but not in its circumferential direction, making the products unsuitable for applications requiring strength in the circumferential direction, such as seals for high-pressure fluids.

[ 0007 ]

Another drawback of an annular seal made of ePTFE is that a fluid flowing through the seal itself creates leakage (hereinafter "permeation leakage") because the seal has a porous structure. To address this problem, it has been proposed in Japanese Laid-Open Patent Application 4-331876 to use an annular seal 35 obtained by applying an outer coat 34 composed of sintered PTFE around the outside of a seal body 33 composed of ePTFE, as shown in Figure 14. In the seal 35, permeation leakage can be prevented by means of the sintered PTFE outer coat 34. However, the two surfaces 35a along which the seal 35 comes into contact with the fastening surface are composed of the hard sintered PTFE outer coat 34, and thus do not adhere well to the fastening surface despite being composed of ePTFE, ultimately failing to prevent interface leakage in a satisfactory manner.

[ 0008 ]

Furthermore, Japanese Laid-Open Patent Application 8-121599 describes a product in which, as shown in Figure 15, the surface layer (hereinafter "the inner peripheral surface") 36b extending around the inside of an annular seal 36 made of ePTFE, that is, the surface in contact with the fluid, is converted to a nonporous molten/fused layer 37 by heating and fusing. Because this seal is composed of ePTFE in areas where it comes into contact with the fastening surface, good bonding with the fastening surface is achieved, and interface leakage is prevented in a satisfactory manner. In addition, the molten/fused layer 37 is used to prevent the fluid from

coming into contact with the portion composed of porous ePTFE, preventing permeation leakage. However, the intrinsic elasticity of ePTFE is ultimately reduced in the circumferential direction because the inner peripheral surface 36b of such an annular seal is composed of the molten/fused layer 37. Another factor is that because the products are manufactured by the same method as conventional seals made of ePTFE, that is, by punching ePTFE sheets, the old drawbacks still remain; namely, ePTFE materials cannot be used efficiently, and the polymer chains are broken by punching. In addition, much labor is involved in forming the molten/fused layers 37 on individual seals obtained by punching.

[ 0009 ]

An object of the present invention, which was devised in view of the above situation, is to provide an easily manufacturable annular seal that forms a tight seal against permeation leakage while retaining the characteristics of ePTFE, that is, the sealing properties of the interface with the fastening surface, and that allows the ePTFE material to be used without creating any waste during fabrication.

[ 0010 ]

[ Means Used to Solve the Above-Mentioned Problems ]

The annular seal pertaining to the present invention is characterized in that a sheet or film made of extended porous polytetrafluoroethylene is structured in multiple layers in the radial direction. An alternative is characterized in that a strip or film made of extended porous polytetrafluoroethylene is wound and laminated spirally or concentrically with the retention of an air core.

[ 0011 ]

It is preferable for at least one fluid permeation inhibitor layer to be formed in the annular seal of the present invention and for the fluid permeation inhibitor layer to be composed of compact polytetrafluoroethylene, and, in particular, of a material obtained by squeezing the pores of extended porous polytetrafluoroethylene. It is also preferable for the aforementioned sheet or film made of extended porous polytetrafluoroethylene to be bonded and laminated by calcining, but it is also permissible to perform bonding and lamination using an adhesive when the material is uncalcined.



[ 0012 ]

A method for manufacturing an annular seal having a fluid permeation inhibitor layer in accordance with the present invention comprises a step in which a strip or film made of extended porous polytetrafluoroethylene is wound and laminated a prescribed number of times, and a step in which a strip or film constituting the fluid permeation inhibitor layer is wound and laminated. An alternative method comprises a step in which a strip or film made of extended porous polytetrafluoroethylene is wound and laminated a prescribed number of times, and a step in which the strip or film made of extended porous polytetrafluoroethylene and obtained by the coating or lamination of a material constituting a fluid permeation inhibitor layer is wound and laminated a prescribed number of time. In an annular seal in which the fluid permeation inhibitor layer is obtained by squeezing the pores of extended porous polytetrafluoroethylene, the aforementioned fluid permeation inhibitor layer composed of compact polytetrafluoroethylene may be formed by squeezing the pores of the aforementioned strip or film made of extended porous polytetrafluoroethylene at least over a distance corresponding to a single turn. In the manufacturing method described above, calcining is performed following the completion of the aforementioned step.

[ 0013 ]

The method for manufacturing an annular seal in accordance with the present invention may also involve a process in which a cylinder is fabricated, and this cylinder is then cut into prescribed lengths in the direction intersecting the longitudinal direction of the cylinder. This cylinder is formed by a process in which a strip or film is wound and laminated helically with the retention of an air core, or a wide strip or film is used to perform winding and lamination spirally or concentrically with the retention of an air core. As used herein, the term "strip or film" refers to a strip or film made of extended porous polytetrafluoroethylene when no fluid permeation inhibitor layer has been laminated, or to a strip or film made of extended porous polytetrafluoroethylene, a strip or film constituting the fluid permeation inhibitor layer, or a strip or film made of extended porous polytetrafluoroethylene and obtained by the coating or lamination of the material constituting the fluid permeation inhibitor layer when the fluid permeation inhibitor layer has been laminated. It is preferable for the resulting cylinder to be cut after being calcined.

[ 0014 ]

[ Embodiments of the Invention ]

Embodiments of the present invention will now be described with reference to drawings.

Figure 1a is a perspective view of the annular seal pertaining to an embodiment of the present invention, and Figure 1b is a longitudinal section of Figure 1a. The annular seal pertaining to the present invention is obtained by spirally winding and laminating a strip or film made of ePTFE (hereinafter collectively referred to as "a film" when no distinction is made between the two), and leaving an air core 10. ePTFE strips 1, 1, ... are bonded and laminated in the radial direction of the seal. It is preferable for the ePTFE strips 1 to have a thickness of 20 to 150  $\mu\text{m}$ , and particularly 50  $\mu\text{m}$  or less. This is because when the thickness exceeds 500  $\mu\text{m}$ , the strips are unsuitable for winding and lamination, and when the thickness is less than 20  $\mu\text{m}$ , handling is impaired, the number of turns increases, and productivity decreases.

[ 0015 ]

As used herein, the term "ePTFE strip" refers to a strip fabricated by a process in which a molding aid is removed from a paste-molded article obtained by mixing the molding aid with a fine PTFE powder, and the strip is then stretched at a high temperature and speed, and calcined as needed. In the case of uniaxial stretching, the nodes (folded crystals) form narrow islands perpendicular to the stretching direction, and fibrils (linear molecular bundles unraveled and drawn out by the stretching of the folded crystals) are oriented in the stretching direction, forming cross-ribs that span the gaps between the nodes. Gaps between the fibrils and spaces defined by the fibrils and the nodes have fibrous structures that serve as air cores. Biaxial stretching yields a spiderweb, fibrous structure in which the fibrils spread out radially, isolated islands are formed from fibril-connecting nodes, and numerous spaces are defined by the fibrils and nodes.

[ 0016 ]

The strip made of ePTFE, which is the material that constitutes the annular seal of the present invention, may be a uniaxially or biaxially stretched ePTFE strip. Although no limitations are imposed on whether calcining is performed following stretching, it is still preferable for an uncalcined ePTFE strip to be used because

uncalcined ePTFE strips adhere better to each other when wound and laminated, and the adhesion is further improved by calcining [the strips] after they have been wound and laminated.

[ 0017 ]

The mean pore diameter of the ePTFE strip constituting the annular seal of the present invention can be adequately set depending on the percent of stretch. It is preferable for the setting to be 0.5–5.0 microns, and particularly 0.5–1.0 micron. This is because excessively large pores will reduce the surface area of contact between the films, reduce adhesion between the films, cause permeation leakage, and compromise rather than improve sealing properties. On the other hand, a mean pore diameter of less than 0.5 micron hampers stretching and fails to produce consistent fiber orientation.

[ 0018 ]

The porosity of the ePTFE strip constituting the annular seal of the present invention can be adequately set at 10–90%, depending on the percent of stretch. It is preferable for the setting to fall within a range of 30 to 85%, depending on the service conditions of the seal (surface roughness, fastening force, and other attributes of fastening members). This is because the seal becomes progressively softer with an increase in porosity, and, while capable of providing a tight seal against a rough surface with a weak fastening force, fails to prevent a substantial permeation leakage from developing.

[ 0019 ]

The annular seal shown in Figure 1 may be manufactured by a direct process in which, as shown in Figure 2, an extended porous polytetrafluoroethylene strip 2 whose width corresponds to the thickness  $t$  of the seal is wound around and laminated on a core 3, or by a process in which an ePTFE cylinder 4 such as that shown in Figure 3 is manufactured by winding and laminating a wide ePTFE strip whose width is several times the thickness  $t$  of the seal, and this cylinder 4 is cut into lengths corresponding to the thickness  $t$  of the seal in the direction intersecting the longitudinal direction of the cylinder 4. The manufacturing of the ePTFE cylinder is not limited to the methods based on the winding of a wide ePTFE strip, and can be accomplished by a method in which, as shown in Figure 4, an ePTFE strip 6 is repeatedly wound in helical fashion

up (Figure 4a) and down (Figure 4b) a core 3, and the assembly is then calcined to form a cylinder 7 (Figure 4c). The upward and downward winding of the strip is not the only alternative. The strip may also be wound in helical fashion by the upward and downward movement of the core 3. The porous nature of the ePTFE strip allows the air trapped during winding to escape through the pores, allowing winding to be accomplished with good adhesion.

[ 0020 ]

It is preferable for the wound article (ring or cylinder) obtained by winding and lamination following the completion of winding to be calcined at a temperature above the melting point of polytetrafluoroethylene, that is, 327 to 380°C, and particularly 350 to 365°C. Calcining the wound article obtained by performing winding while avoiding air entrapment causes the ePTFE film to shrink somewhat as a result of a heat treatment, bonding and integrating laminated strips to the extent where the overlapping portions are virtually indistinguishable. Calcining is performed at 380°C or lower in order to prevent the pores from being destroyed by heat fusion. It is preferable for the calcining to be performed while the core 3 is inserted into the assembly, and the core 3 should be taken out following calcining.

[ 0021 ]

A method in which ePTFE films are bonded to each other with the aid of an adhesive can be used to bond and laminate the films to each other when these films are not calcined. PTFE ordinarily has low adhesiveness, but the fibrous structure of ePTFE films allows the adhesive to penetrate into the pores, bonding the films together due to the anchor effect. Although the annular seal shown in Figure 1 was obtained by the spiral winding and lamination of ePTFE strips, the annular seal of the present invention may also be obtained by concentric winding and lamination. The cylinder shown in Figure 3 may also be fabricated by spiral or concentric winding and lamination.

[ 0022 ]

The annular seal of the present invention can be conferred with various thicknesses  $t$  by varying the width  $a$  of the ePTFE strip in the manufacturing method shown in Figure 2 or by varying the cutting width  $t$  of the cylinder 4 in the manufacturing method shown in Figure 3. In addition, annular seals having various

inside diameters  $d$  can be obtained by varying the outside diameter of the core 3, and seals having various outside diameters  $D$ , which correspond to the size of the fastening members, can be obtained by varying the thickness of the ePTFE strip or the number of turns. The annular seal of the present invention is therefore free from the size limitations imposed in the past in order to maintain the desired ePTFE characteristics, as in the case of conventional extrusion molding, making it possible to provide seals of varying sizes. In particular, seals having the following dimensions are suitable. Specifically, the inside diameter  $d$  can be appropriately selected from within a range of 10–200 mm because of the size of the core 3, and the outside diameter  $D$  can be appropriately selected from within a range of 15–300 mm because of considerations associated with the type of core 3 and the weight of the wound article. In addition, the thickness  $t$  of the annular seal is commonly 0.5 to 10 mm.

[ 0023 ]

It is preferable for the core used in accordance with the manufacturing method described above to be heat-resistant and readily deformable. Specific examples include circular rods or cylinders made of copper, stainless steel, or heat-resistant plastics (aramids, polyimides, and the like).

[ 0024 ]

It is preferable for such a core to be coated with a release agent or covered with a release sheet around the outer periphery of the core in order to facilitate the removal of the core or the ePTFE cylinder following calcining. The joint use of a release agent or release sheet is unnecessary if the core itself can be removed easily. Examples of easily removable cores include split-structure cores that can be split and taken out following calcining, and cores whose surfaces have been roughened to imitate a stainless mesh. Another example is a core in which the surface around the outside of the cylinder has been provided with a large number of small holes 8. Such a core can be removed easily because water or air can be blown into the interior 9 of the cylindrical core following the completion of winding, reducing the adhesion between the core and the wound article.

[ 0025 ]

The core shape is not limited to the round rod and includes various rod shapes such as those that have elliptical, square, and other polygonal cross sections. Seals that

are other than annular in shape may also be manufactured for some core shapes. Figure 6 shows an annular seal manufactured using a square core. A core shaped as a truncated cone whose outside diameter gradually decreases can also be used in the case of a manufacturing method in which ePTFE strips are helically wound upward or downward (see Figure 4). This arrangement yields an annular seal whose inside diameter gradually increases, that is, an annular seal that can be used between fastening members of varying diameters.

[ 0026 ]

ePTFE materials are used without creating any waste because the annular seal of the present invention can be manufactured in a manner such as that described above. Specifically, manufacturing involves winding and laminating ePTFE strips, so no material is wasted and the production is more economical than the conventional manufacturing method in which sheets are punched.

[ 0027 ]

In addition, the ePTFE annular seal thus manufactured is soft due to the characteristics of the ePTFE, improving conformity with the irregularities of the fastening surface and making it possible to achieve good adhesion to the fastening surface with a weak fastening torque and to prevent interface leakage. The annular seal of the present invention can be manufactured in a variety of thicknesses by varying the width of the strips to be wound and laminated, and can thus be used as a seal for a mating pipe connection such as that shown in Figure 7 or as a seal for a shaft part such as shown in Figure 8. The reason is that when the product is used as a seal for a mating pipe connection such as that shown in Figure 7, the softness of the ePTFE allows the pipe 20 on the female end to be pressed firmly against the pipe 21 on the male end, making it possible to prevent interface leakage in proportion to the improved conformity of the two pipes with the irregularities of the fastening surfaces 20a and 21a, and affording higher corrosion resistance than with rubber sealants. Another reason is that, as shown in Figure 8, the excellent frictional and dust- and drip-proofing properties of PTFE can be preserved in seals provided to components in which dust is to be prevented from penetrating inside from the outside of a casing 24 containing an inserted shaft 23, or to components in which oil is to be prevented from being spilled out from lubricated portions. This approach can also be used with the seal of a cover member 25, as shown in Figure 9. Yet another reason is that because an annular seal

made of ePTFE is soft, the bottomed cylindrical member 26 can be made airtight by securely fastening the assembly along threaded sections. In Figures 7 through 9, 22 is an annular seal.

The annular seal pertaining to another embodiment of the present invention will now be described with reference to Figure 10.

[ 0028 ]

In this annular seal, a fluid permeation inhibitor layer 12 is interposed between ePTFE layers 11a and 11b, which are obtained by the winding and lamination of ePTFE strips. The fluid permeation inhibitor layer 12 is designed to prevent the fluid that has passed through the ePTFE layer 11a around the inner periphery from reaching the ePTFE layer 11b around the outer periphery, and is composed of a material that is devoid of pores in order to prevent fluid permeation. Examples of materials for constructing the fluid permeation inhibitor layer 12 include nonporous PTFE (hereinafter referred to as "compact PTFE"), resins other than PTFE (hot-melt resins, thermosetting resins), silicone rubber and other rubber materials, and metals. These materials can be appropriately selected in accordance with the type of environment in which the seal is used (in particular, the type of liquid flowing through the piping system), the method used for seal manufacture (in particular, the presence or absence of calcining), the characteristics to be obtained, and the like. For example, it is preferable to use a strip made of compact PTFE with high corrosion resistance when a corrosive fluid is to be sealed. A metal strip (metal foil) may be used when a high-pressure fluid is involved. Examples of strips made of compact PTFE include strips consisting of sintered PTFE and strips obtained by a process in which a plurality of ePTFE strips are superposed, and the pores in ePTFE are then squeezed. Strips composed of compact PTFE manufactured by the squeezing of ePTFE pores are suitable for the material constituting the fluid permeation inhibitor layer, which must be resistant to corrosion, because the pores are squeezed while the fiber orientation of ePTFE is kept the same.

[ 0029 ]

The fluid permeation inhibitor layer 12 may be composed of a single strip or foil or may be obtained by the monolithic lamination of a plurality of strips. In effect, the strips for forming a single fluid permeation inhibitor layer 12 can have any

thickness as long as they remain flexible enough to be wound and laminated with reasonable ease and as long as adequate adhesion can be achieved between the ePTFE layers 11a and 11b. It is therefore preferable for the thickness of the fluid permeation inhibitor layer 12 to be between 5 and 500  $\mu\text{m}$ , and particularly between 50 and 200  $\mu\text{m}$ , depending on the type of material constituting the fluid permeation inhibitor layer.

[ 0030 ]

When the fluid permeation inhibitor layer 12 is composed of a resin other than PTFE, it is possible to form the fluid permeation inhibitor layer 12 by heating and curing a high-viscosity liquid or an emulsion solution such as that obtained by curing a thermosetting resin to the B-state, or by solidifying a hot-melt resin through cooling. In view of considerations related to the manufacturing method described above, the thickness of the fluid permeation inhibitor layer 12 formed in this case is limited to the range that permits coating operations to be conducted.

[ 0031 ]

The position of the fluid permeation inhibitor layer 12 is not limited to that between the ePTFE layers, as shown in Figure 10, and may further include positions around the inner or outer peripheral surfaces of the seal. To achieve better adhesion, however, it is preferable [for the fluid permeation inhibitor layer] to be interposed between the ePTFE layers.

[ 0032 ]

Fluids are prevented from penetrating into the annular seal when the fluid permeation inhibitor layer constitutes the innermost layer, and fluids that have permeated the ePTFE layers are prevented from leaking out to the outside of the seal when [the fluid permeation inhibitor layer] constitutes the outermost layer. A fluid that has penetrated from the outside can be prevented from entering the piping system at any position of the fluid permeation inhibitor layer. In addition, most of the area in contact with the fastening surface is composed of ePTFE, making it possible for the ePTFE seal to adequately prevent interface leakage while preserving its conformity with the fastening surface.



[ 0033 ]

Although only one fluid permeation inhibitor layer is interposed in the annular seal shown in Figure 10, it is also possible to provide the annular seal of the present invention with a plurality of interposed fluid inhibitor layers. Figure 11 shows a seal containing two interposed fluid permeation inhibitor layers. The following components are then laminated in order from inside outward: an ePTFE layer 13a, a fluid permeation inhibitor layer 14a, an ePTFE layer 13b, a fluid permeation inhibitor layer 14b, and an ePTFE layer 13c. In a seal thus configured, the fluid permeation inhibitor layer 14b around the outer peripheral surface can prevent a fluid from flowing outside the seal even when this fluid, after penetrating the ePTFE layer 13a around the inner periphery, has passed through the fluid permeation inhibitor layer 14a around the inner peripheral surface and is about to enter the intermediate ePTFE layer 13b. Sealing properties are thus improved as the number of interposed fluid permeation inhibitor layers increases.

[ 0034 ]

An annular seal containing such interposed fluid permeation inhibitor layers is manufactured by appropriately combining a step in which an ePTFE film is wound and laminated a prescribed number of times (step A), and a step in which a strip constituting a fluid permeation inhibitor layers is wound and laminated (step B).

[ 0035 ]

For example, the steps should be continuously performed in the sequence A, B, A, for the annular seal shown in Figure 10, and in the sequence A, B, A, B, A for the annular seal shown in Figure 11. The fluid permeation inhibitor layers have lower adhesiveness than the ePTFE layers, but sandwiching the fluid permeation inhibitor layers between the ePTFE layers makes it possible to achieve good bonding without the use of an interposed adhesive layer or the like. Therefore, seal fabrication is accomplished in the following manner: a seal in which a fluid permeation inhibitor layer is sandwiched between ePTFE layers is first fabricated when the fluid permeation inhibitor layer constitutes the innermost or outermost layer, and the innermost ePTFE layer is then peeled off when the fluid permeation inhibitor layer constitutes the innermost layer, or the outermost ePTFE layer is peeled off when the fluid permeation inhibitor layer constitutes the outermost layer.

[ 0036 ]

In addition, the following two options exist for manufacturing an annular seal in which a fluid permeation inhibitor layer is composed of compact polytetrafluoroethylene: ① the strip used in the aforementioned step B is composed of compact polytetrafluoroethylene and is obtained by superposing a plurality of ePTFE films that are separate from the ePTFE strips used for winding and lamination, and squeezing the pores, and ② components are wound and laminated after part of an ePTFE strip has been converted to compact polytetrafluoroethylene by squeezing the pores of the ePTFE strip along a section corresponding to the fluid permeation inhibitor layer of the spirally wound and laminated ePTFE strip. In ①, every time a fluid permeation inhibitor layer is interposed, it is necessary to halt the winding and lamination of the ePTFE strip (step A) and to switch to the winding and lamination of the compact polytetrafluoroethylene strip constituting the fluid permeation inhibitor layer (step B), whereas manufacturing method ② allows the procedure to be completed in one step throughout the entire winding process.

[ 0037 ]

In any of these manufacturing methods, it is preferable for the system to be calcined at the melting point of ePTFE, that is, at 327 to 380°C, and especially 350 to 365°C, following the completion of the winding and lamination step. The calcining-induced shrinkage of the ePTFE strips makes it possible to improve adhesion between the ePTFE strips and between the ePTFE strips and the fluid permeation inhibitor layer.

[ 0038 ]

When a fluid permeation inhibitor layer is formed by the curing of a high-viscosity liquid or solution or by the cooling and solidification of a hot-melt resin, an ePTFE strip obtained by the coating or lamination of materials constituting fluid permeation inhibitor layers is wound and laminated in advance during the step for winding and laminating the ePTFE strip. An operation in which the material constituting the fluid permeation inhibitor layer is applied by a coater or the like to the area of the ePTFE strip corresponding to the position in which the fluid permeation inhibitor layer is interposed facilitates operations involved in the application of the resin for forming the fluid permeation inhibitor layer and makes it possible to manufacture

an annular seal in which a fluid permeation inhibitor layer is interposed in an appropriate position. The ePTFE film is commonly bonded with the aid of an adhesive if the material constituting the fluid permeation inhibitor layer does not allow calcining to be performed at a high temperature. When an ePTFE strip is to be bonded and laminated with the aid of an adhesive, it is preferable for the ePTFE strip to be coated with an adhesive before being bonded and laminated.

[ 0039 ]

Similar to the method for manufacturing an annular seal devoid of a fluid permeation inhibitor layer, the method for manufacturing an annular seal having such a fluid permeation inhibitor layer may be accomplished by a process in which a strip whose width corresponds to the thickness  $t$  of the seal is used to directly manufacture an annular seal having a fluid permeation inhibitor, or by a process in which a wide strip is used to form a cylinder having a fluid permeation inhibitor layer, and this cylinder is cut in the direction intersecting the longitudinal direction of the cylinder, producing seals having a thickness  $t$  (see Figure 3). The cylinder having a fluid permeation inhibitor layer may also be fabricated by winding and laminating a strip in helical fashion (see Figure 4). For reasons identical to those described with reference to the direct method for manufacturing an annular seal, it is preferable for the cylinder thus fabricated to be cut up after being calcined under the same conditions.

[ 0040 ]

Such manufacturing methods allow the product to be continuously manufactured using a simple winding step despite the presence of a fluid permeation inhibitor layer. The entire winding process can thus be automated, making it possible to manufacture an annular seal whose sealing characteristics are the same as, or better than, the sealing characteristics of a product obtained by a method in which an ePTFE seal body is coated with a PTFE outer coat or the inner peripheral surface of an ePTFE annular seal is fused to manufacture a fused and solidified layer. It is apparent that the manufacturing method involving the winding and laminating of a strip allows materials to be used more efficiently than in the case of a conventional manufacturing method based on sheet punching and that this method has the same merits as those described above in relation to economic efficiency.

[ 0041 ]

[ Embodiments ]

The present invention will now be described through specific embodiments.

Embodiment 1

A film was formed from a resin paste obtained by mixing 22 weight parts of solvent naphtha with 100 weight parts of a polytetrafluoroethylene powder (fine powder) obtained by emulsion polymerization, the resulting paste-molded article in the shape of a film was heated to a temperature above the boiling point of the solvent naphtha to evaporate off the solvent naphtha, and the article was then biaxially stretched at a rate of at least 10% per second and a temperature below the melting point of polytetrafluoroethylene, yielding an ePTFE film with a thickness of 60  $\mu$ m and a porosity of 80%.

[ 0042 ]

A core wrapped in a stainless steel mesh as a release material was prepared in the form of a solid steel bar with a diameter of 31 mm and a length of 400 mm. The release material was temporarily tacked to the ends of the wrapped section with an adhesive tape.

[ 0043 ]

A film fabricated as described above was spirally wound and laminated on this core. After 70 turns had been formed, the film ends were cut off with a cutter and smoothed down and fixed to the wound article to prevent the cut ends of the ePTFE film from being rolled up. The adherence of film sections to each other under pressure was facilitated by a procedure in which the wound article obtained by completing the winding and lamination of the ePTFE film was rolled on a table to create pressure. Following such bonding under pressure, the wound article was placed in an oven and calcined for 75 minutes at 353°C. Following calcining, the wound article was taken out of the oven and cooled by being dipped in a water bath. The core was pulled out after [the wound article] had been cooled to room temperature, yielding a cylinder in which the ePTFE layers were calcined and integrated. Annular seals were obtained by cutting the cylinder with a slicer into 1.5-mm widths in the direction intersecting the longitudinal direction. The resulting seals had an inside diameter of 31 mm, an outside

diameter of 34.5 mm, a thickness of 1.5 mm, and a density of 1.1 g/cm<sup>3</sup>. It was also found that the seals shrunk due to calcining. On the other hand, the strip could be peeled off from the portions of the resulting annular seals where winding had been completed, indicating that the ePTFE films were not integrally fused.

[ 0044 ]

Embodiments 2 and 3

The ePTFE film fabricated in Embodiment 1 was used to fabricate annular seals in the same manner as in Embodiment 1 except that the type of release material for the core, the number of turns, the calcining time, and the presence or absence of bonding under pressure were varied, as shown in Table 1. The outside diameters and densities of the resulting annular seals are shown in Table 1, indicating that shrinkage was induced by calcining in both cases.

[ 0045 ]

Embodiment 4

A uniaxially extended ePTFE film (thickness: 125  $\mu$ m) was fabricated from a PTFE resin paste prepared in accordance with Embodiment 1. Annular seals were fabricated in the same manner as in Embodiment 1 under the conditions shown in Table 1.

[ 0046 ]

[ Table 1 ]

Emboilment	ePTFE film		Manufacturing conditions					Seal	
	Stretching	Thickness (μm)	Release material	Number of turns	Bonding under pressure	Calcining time (min)	Cooling method	Outside diameter (mm)	Density (g/mm)
1	Biaxial	60	SUS	70	Performed	75	Water cooling	34.5	1.1
2	Biaxial	60	Aramid	105	Performed	80	Water cooling	37.0	1.0
3	Biaxial	60	Aramid	75	Not performed	80	Water cooling	35.5	0.95
4	Uniaxial	125	Aramid	30	Not performed	45	Air cooling	38.0	1.3

[ 0047 ]

Embodiment 5

A film was formed from a resin paste obtained by mixing 22 weight parts of solvent naphtha with 100 weight parts of a polytetrafluoroethylene powder (fine powder) obtained by emulsion polymerization, the resulting paste-molded article in the shape of a film was heated to a temperature above the boiling point of the solvent naphtha to evaporate off the solvent naphtha, and the article was then biaxially stretched at a rate of at least 10% per second and a temperature below the melting point of polytetrafluoroethylene, yielding an ePTFE film with a thickness of 60  $\mu\text{m}$  and a porosity of 80%.

[ 0048 ]

A core wrapped in a stainless steel mesh as a release material was prepared in the form of a solid steel bar with a diameter of 31 mm and a length of 400 mm. The release material was temporarily tacked to the ends of the wrapped section with an adhesive tape.

[ 0049 ]

A film fabricated as described above was spirally wound and laminated on this core. After 50 turns had been formed, the film ends were cut off with a cutter and smoothed down and fixed to the wound article to prevent the cut ends of the ePTFE film from being rolled up. After nine turns of the ePTFE film had subsequently been laid on top of each other, the pores were squeezed, and a single turn of the resulting compact PTFE strip was wound. Fifty turns of the ePTFE film were then rewound, the film was cut off with a cutter, and the ends were smoothed down and fixed to the roll to prevent rolling up. The adherence of film sections to each other under pressure was facilitated by a procedure in which the wound article thus obtained was rolled on a table to create pressure. Following such bonding under pressure, the wound article was placed in an oven and calcined for 75 minutes at 353°C. Following calcining, the wound article was taken out of the oven and cooled by being dipped in a water bath. The core was pulled out after [the wound article] had been cooled to room temperature, yielding a cylinder which had a fluid permeation inhibitor layer and in which the ePTFE layers were calcined and integrated. Annular seals were obtained by cutting the

cylinder with a slicer into 1.5-mm widths in the direction intersecting the longitudinal direction. The seals had an inside diameter of 31 mm, an outside diameter of 37 mm, and a thickness of 1.5 mm. The density of the resulting seals was 1.1 g/cm<sup>3</sup>.

[ 0050 ]

Comparative Example 1

A biaxially extended PTFE film with a porosity of 82% and a thickness of 55  $\mu$ m was obtained using a resin paste prepared in accordance with Embodiment 5. This film was laminated 40 times and was integrated by being calcined at 365°C, yielding an ePTFE sheet with a porosity of 73% and a thickness of 1.5 mm. This sheet was punched out, yielding annular seals with an inside diameter of 31 mm, an outside diameter of 37 mm, and a thickness of 1.5 mm.

[ 0051 ]

Comparative Example 2

A commercially available sintered polytetrafluoroethylene sheet with a thickness of 1.5 mm was punched out with a die, yielding annular seals consisting of sintered PTFE and having an inside diameter of 31 mm, an outside diameter of 37 mm, and a thickness of 1.5 mm.

[ 0052 ]

Evaluation

The following evaluation method was used as a basis for finding the relation between the fastening force and the amount of leakage for the annular seals fabricated in Embodiment 5 and in Comparative Examples 1 and 2. The fastening members used had fastening surfaces with surface roughness levels of 8 S and 100 S.

[ 0053 ]

As shown in Figure 12a, a resulting annular seal 16 was set in the upper opening of a bottomed cylinder 17, which was then covered with a lid 18. The pressure inside the container 17 was raised to 5 atm by blowing compressed air [into the container while it was] closed with the lid (Figure 12b). The fastening pressure was gradually raised, and the amount of leakage was measured at 50 and 100 kgf/cm<sup>2</sup>. Here, the fastening pressure was adjusted by varying the load applied to the lid 18.



For example, the fastening pressure of 50 kgf/cm<sup>2</sup> was obtained by applying a load of 160 kgf, and the fastening pressure of 100 kgf/cm<sup>2</sup> was obtained by applying a load of 320 kgf. The amount of leakage was determined in the following manner: a gage was used to read the internal pressure of the container 12 [sic]  $t$  seconds after the cock had been closed, and the result was calculated using the formula  $P \times 50/t$  (unit: atm · cc/sec), where  $P$  is the reduction in internal pressure (unit: atm). In the formula, "50" is the volume (cc) of the portion containing trapped air. The results are shown in Table 2.

[ 0054 ]

[ Table 2 ]

Roughness of fastening surface	8 S		100 S	
	50 kg/cm <sup>2</sup>	100 kg/cm <sup>2</sup>	50 kg/cm <sup>2</sup>	100 kg/cm <sup>2</sup>
Embodiment 5	0.0004 atm · cc/sec	—	0.25 atm · cc/sec	0.0052 atm · cc/sec
Comparative Example 1	0.76 atm · cc/sec	0.093 atm · cc/sec	0.83 atm · cc/sec	0.086 atm · cc/sec
Comparative Example 2	0.015 atm · cc/sec	0.0060 atm · cc/sec	Unmeasurable	5.1 atm · cc/sec

[ 0055 ]

The major drawback of a rough fastening surface (100 S) is interface leakage. Because a sintered PTFE seal (Comparative Example 2) allows considerable interface leakage, measurement is impossible when the fastening force is weak (50 kg/cm<sup>2</sup>). By contrast, ePTFE seals (Embodiment 5, Comparative Example 1) conform well to rough surfaces and can prevent interface leakage, and thus have better sealing properties than do sintered PTFE seals both at a fastening force of 50 kg/cm<sup>2</sup> and at a fastening force of 100 kg/cm<sup>2</sup>. In addition, the seal of Embodiment 5 has better sealing properties than does the ePTFE seal manufactured by punching (Comparative Example 1).

[ 0056 ]

On the other hand, interface leakage is unlikely to occur at a smooth fastening surface (8 S), so the leakage properties are improved for a sintered PTFE seal (Comparative Example 2) but remain virtually unchanged for a PTFE sheet seal devoid of a fluid permeation inhibitor layer (Comparative Example 1). In an ePTFE seal having a fluid permeation inhibitor layer (Embodiment 5), a smooth surface affords

better sealing properties than a rough surface does, and leakage cannot be measured at a fastening pressure of 100 kg/cm<sup>2</sup> because virtually leakage exists. It was thus learned that a fluid permeation inhibitor layer can form a tight seal against permeation leakage.

[ 0057 ]

[ Merits of the Invention ]

The present invention involves manufacturing an annular seal by the winding and lamination of an ePTFE strip, making it possible to manufacture annular seals of various sizes without wasting any material. This approach can be applied to a wide variety of seal applications because ePTFE characteristics afford adequate softness and excellent corrosion resistance.

[ 0058 ]

In addition, an annular seal having a fluid permeation inhibitor layer can have excellent sealing properties because ePTFE characteristics allow the seal to adhere even to rough fastening surfaces, preventing interface leakage, and because the fluid permeation inhibitor layer is capable of preventing permeation leakage.

[ 0059 ]

Another merit of the proposed method for manufacturing an annular seal having a fluid permeation inhibitor layer is that seals of various sizes can be manufactured without any loss of material merely by automating the operations of winding and lamination, despite the presence of a fluid permeation inhibitor layer.

[ Brief Description of the Drawings ]

[ Figure 1 ]

A diagram depicting the structure of an embodiment of the annular seal of the present invention.

[ Figure 2 ]

A diagram illustrating a method for manufacturing the annular seal depicted in Figure 1.

[ Figure 3 ]

A diagram illustrating another method for manufacturing the annular seal depicted in Figure 1.

**[ Figure 4 ]**

A diagram illustrating yet another method for manufacturing the annular seal depicted in Figure 1.

**[ Figure 5 ]**

A diagram depicting an embodiment of the core used in the manufacture of the annular seal of the present invention.

**[ Figure 6 ]**

A diagram depicting the structure of another embodiment of the annular seal of the present invention.

**[ Figure 7 ]**

A diagram illustrating the manner in which an annular seal is used.

**[ Figure 8 ]**

A diagram illustrating the manner in which an annular seal is used.

**[ Figure 9 ]**

A diagram illustrating the manner in which an annular seal is used.

**[ Figure 10 ]**

A diagram depicting the structure of an embodiment of the annular seal containing a fluid permeation inhibitor layer and pertaining to the present invention.

**[ Figure 11 ]**

A diagram depicting the structure of another embodiment of the annular seal containing a fluid permeation inhibitor layer and pertaining to the present invention.

**[ Figure 12 ]**

A diagram illustrating a method for measuring the relation between fastening pressure and amount of leakage.

**[ Figure 13 ]**

A diagram illustrating the drawbacks of a conventional annular seal.

**[ Figure 14 ]**

A diagram depicting the structure of a conventional annular seal.

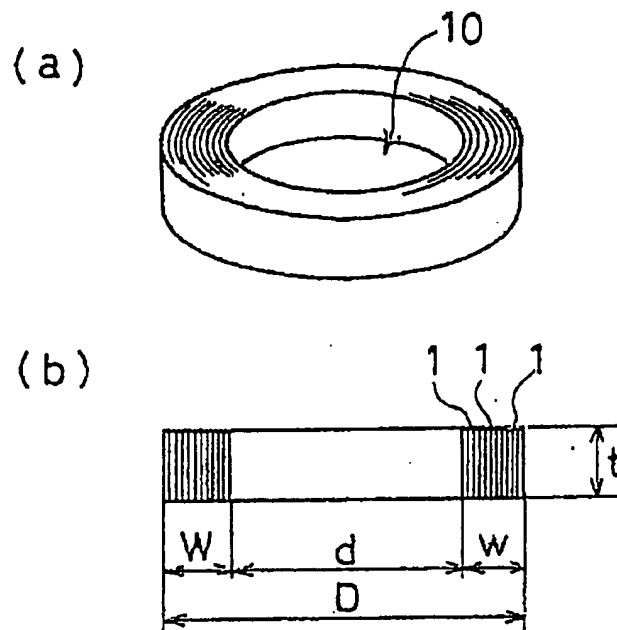
[ Figure 15 ]

A diagram depicting the structure of a conventional annular seal.

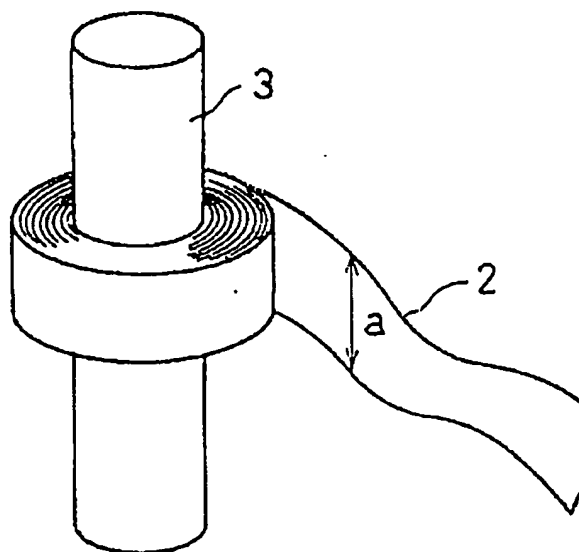
[ Key to Symbols ]

1:	ePTFE strip
2:	ePTFE strip
3:	core
6:	ePTFE strip
11a, 11b:	ePTFE strips
12:	fluid permeation inhibitor layer
13a, 13b, 13c:	ePTFE layers
14a, 14b:	fluid permeation inhibitor layers
22:	annular seal

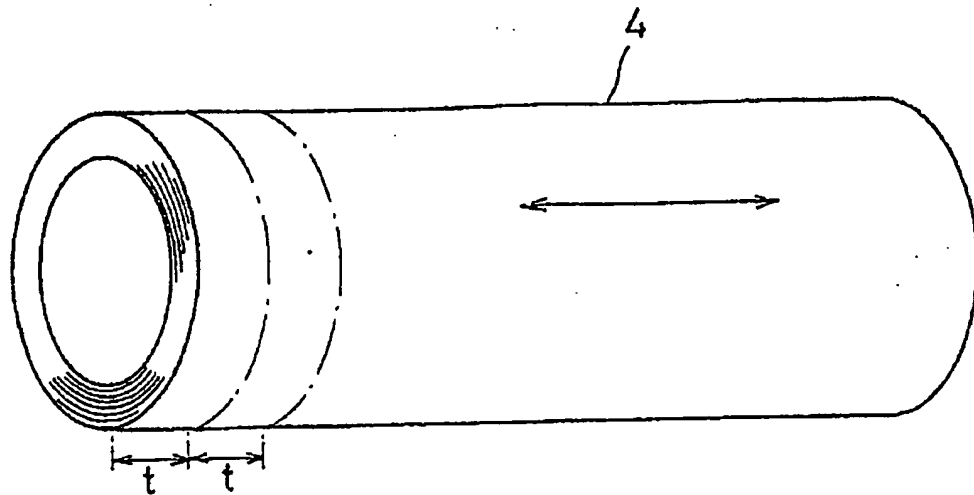
[ Figure 1 ]



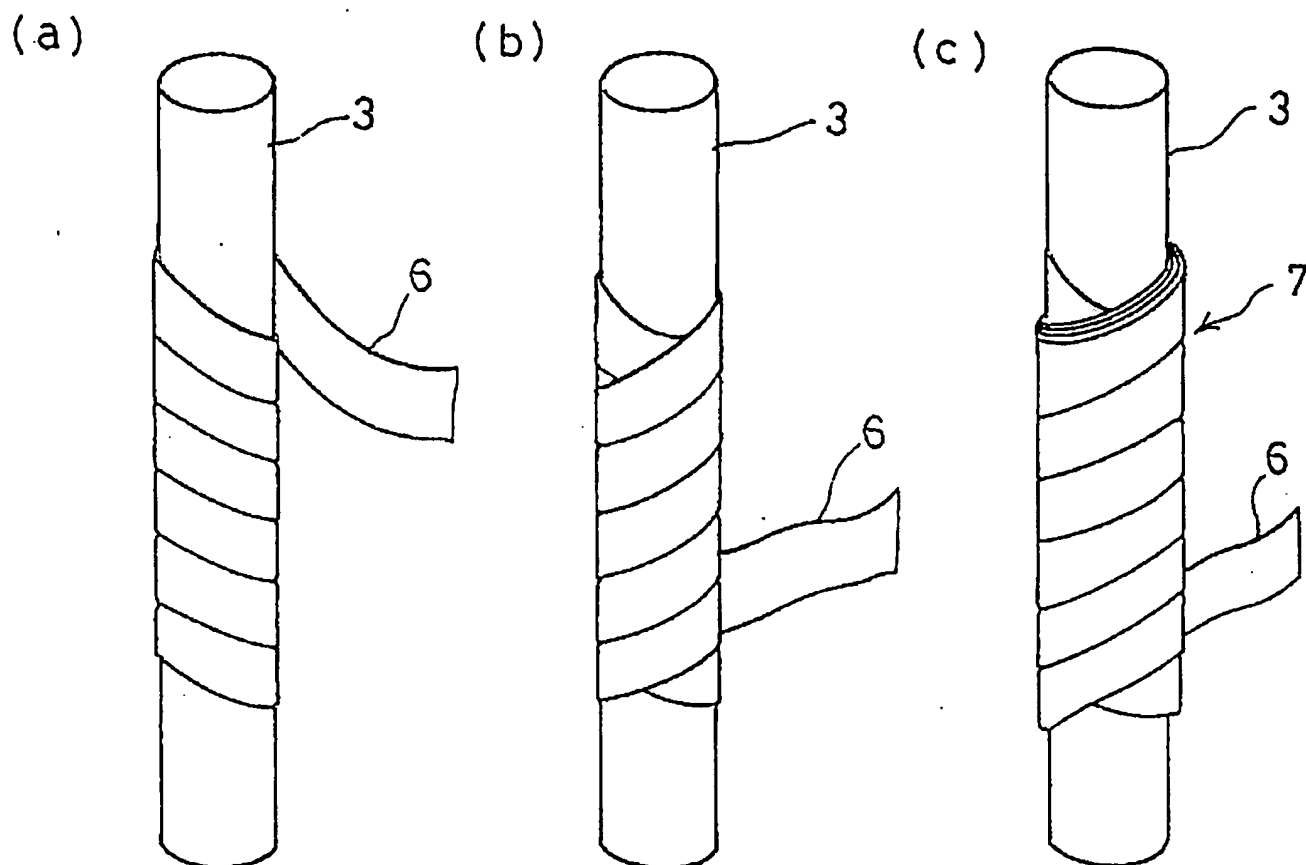
[ Figure 2 ]



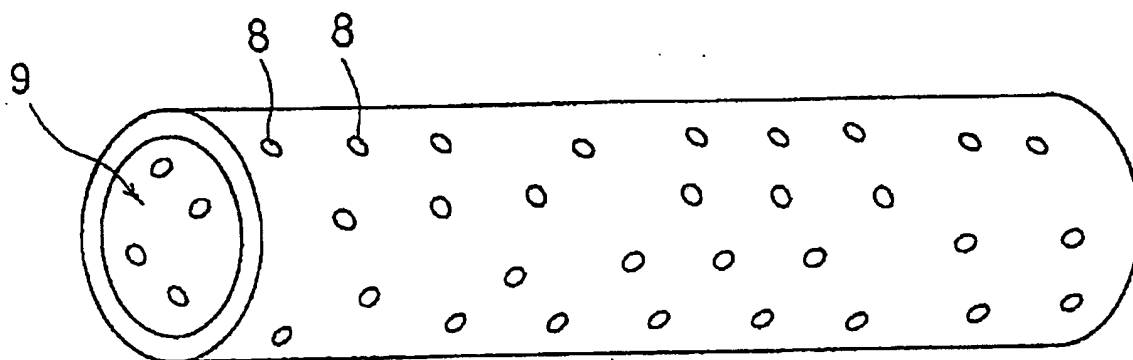
[ Figure 3 ]



[ Figure 4 ]

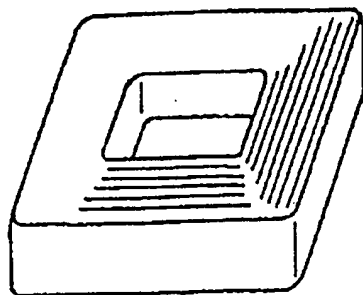


[ Figure 5 ]

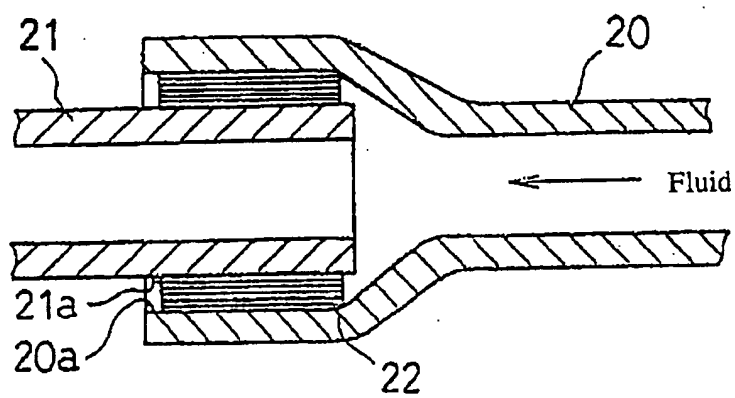




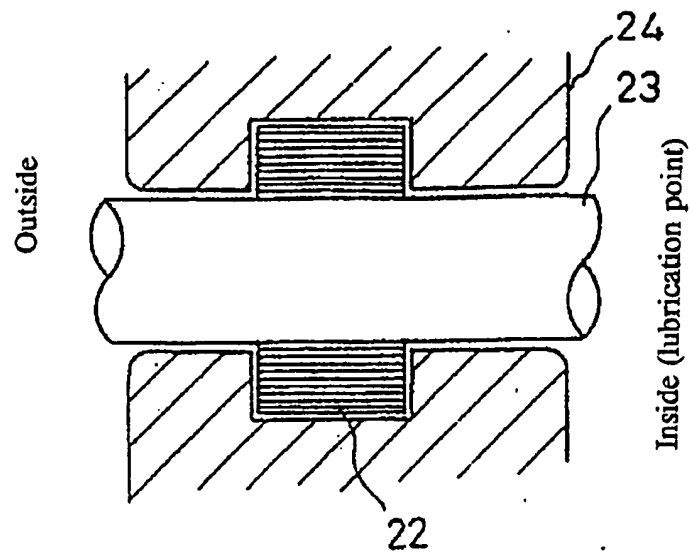
[ Figure 6 ]



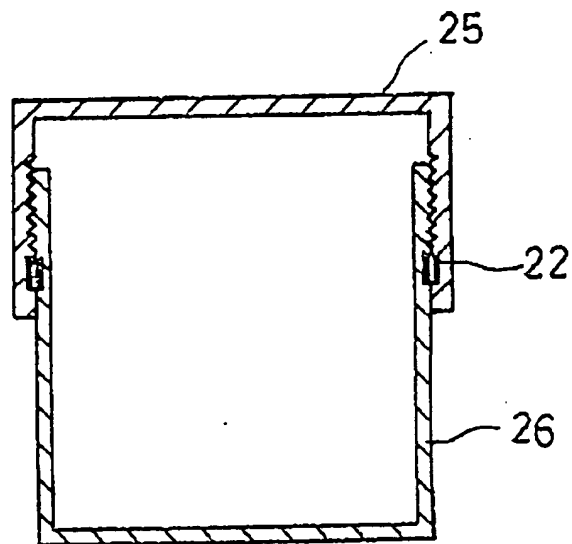
[ Figure 7 ]



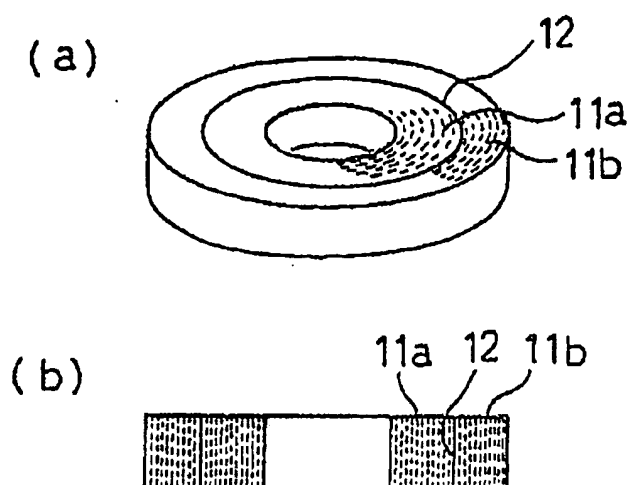
[ Figure 8 ]



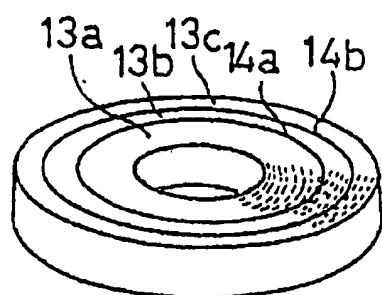
[ Figure 9 ]



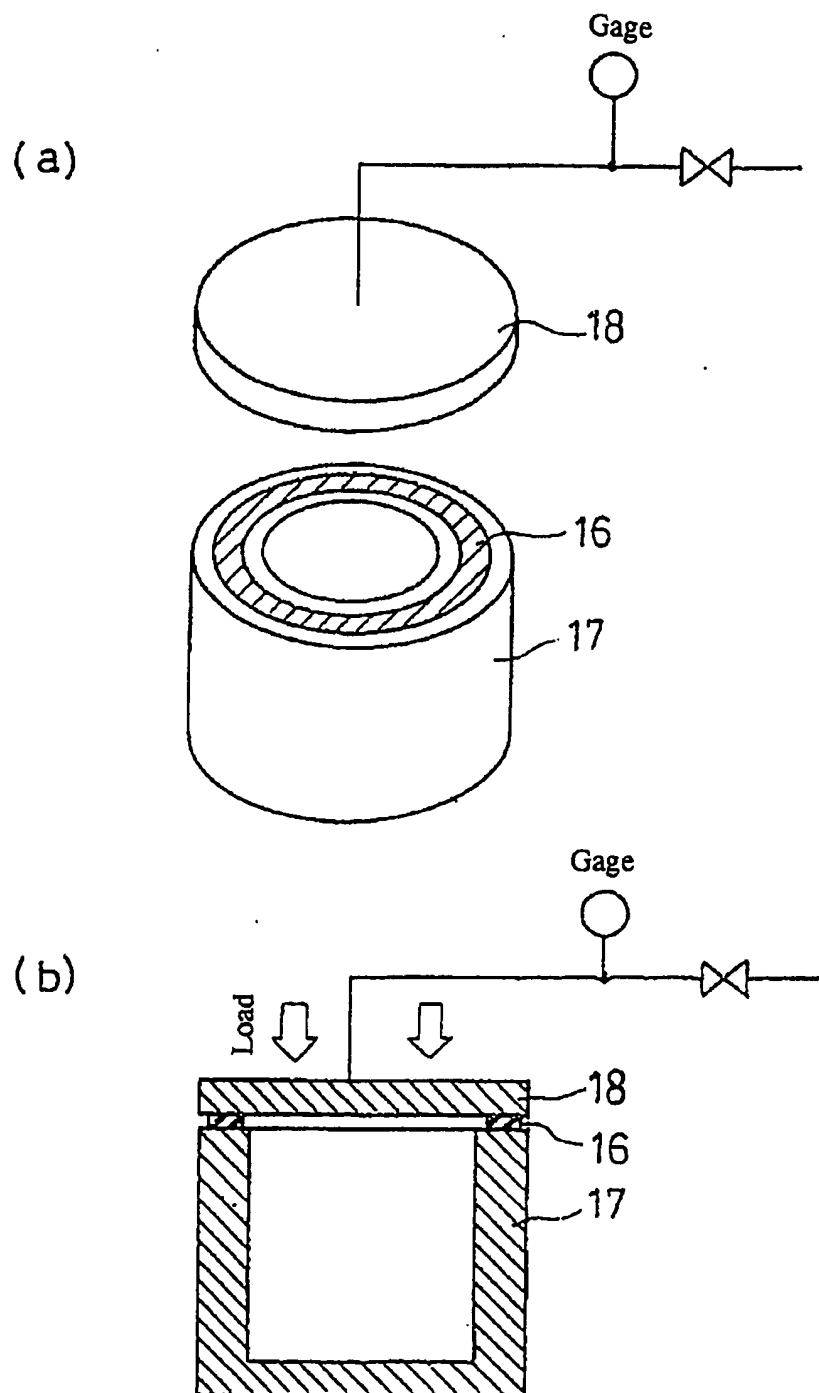
[ Figure 10 ]



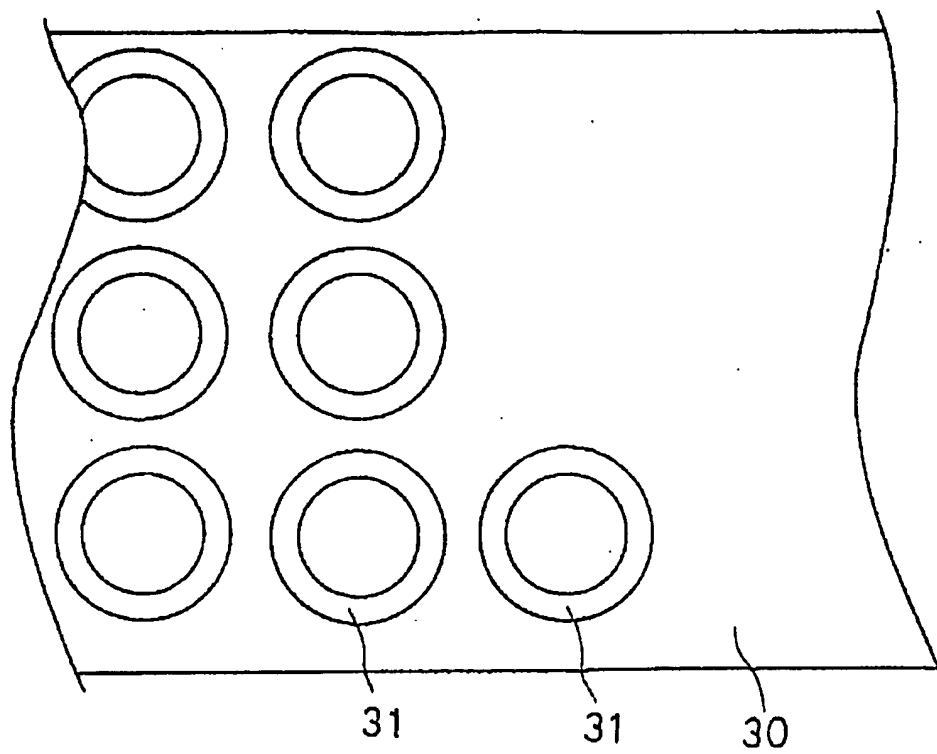
[ Figure 11 ]



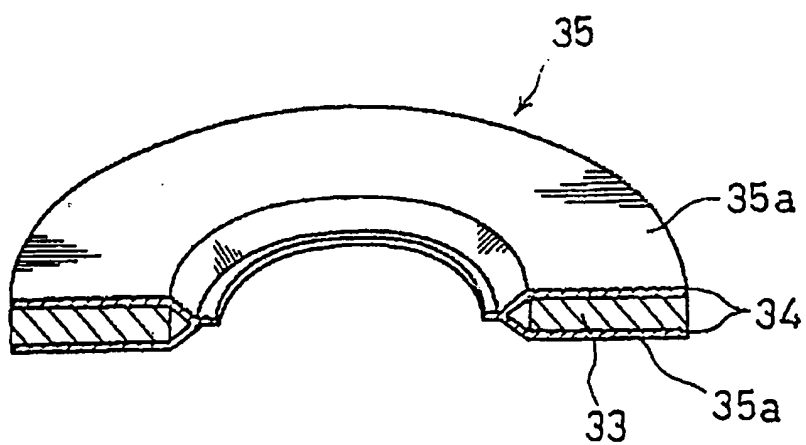
[ Figure 12 ]



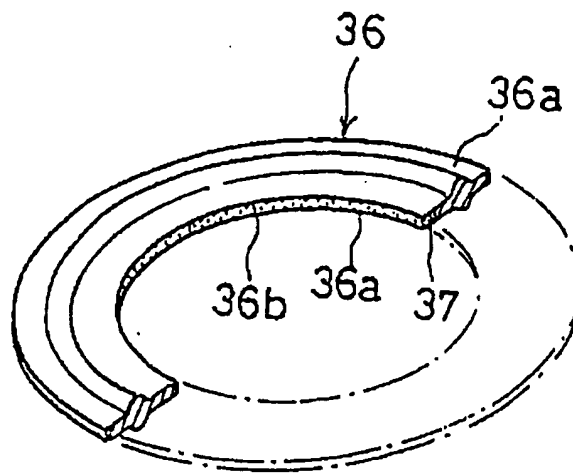
[ Figure 13 ]



[ Figure 14 ]



[ Figure 15 ]



[ Document Title ]    Abstract

[ Abstract ]

[ Object ]    To provide an easily manufacturable annular seal in which the ePTFE material can be used without creating any waste during manufacture and which forms a tight seal against permeation leakage without adversely affecting the characteristics of extended porous polytetrafluoroethylene (ePTFE), that is, without adversely affecting the sealing properties along the interface with the fastening surface; and to provide a method for manufacturing such a seal.

[ Means ]    A seal in which an ePTFE sheet or film is structured in multiple layers in the radial direction and which is preferably provided with at least one fluid permeation inhibitor layer composed of compact PTFE. This seal is manufactured by a process in which an ePTFE strip is wound and laminated spirally or concentrically with the retention of an air core, and a strip constituting a fluid permeation inhibitor layer is wound and laminated as needed.

[ Selected Figure ]    Figure 1

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